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(54) A PROCESS FOR THE PREPARATION OF POWDERED MALTITOL

(71) We, KOWA COMPANY LTD., a Corporation organized and existing under the laws of Japan of 6-29 Nishiki 3-chome, Naka-ku, Nagoya, Japan, do hereby declare the invention for which we pray that a patent may be granted to us and the method by which it is to be performed to be particularly described in and by the following statement:—

This invention relates to a process for the preparation of powdered maltitol. Maltitol [4 - (α - D - glucopyranosyl) - D - sorbitol] is a disaccharide alcohol which has a sweetness of 85 to 95% of sucrose, and is recommended as sweetening agent for diabetic food (see, for example, British Patent Specification No. 1,250,952) in a similar manner to sorbitol or levulose. It is, however, recognized as being non-decomposable in intestinal tracts and also as being non-utilizable *in vivo*, and is, therefore, different from the other sweetening agents. Therefore, it may be also recommended as a sweetening agent in low-calorie diets.

Maltitol is also different from the other sweetening agents such as sorbitol, levulose, dextrose and sucrose, in that it is amorphous and very hygroscopic in the solid state, and it is difficult to obtain free flowing powder from the aqueous solution by conventional drying processes. Even if it is dried completely, the mass obtained changes to jelly or to a liquid state in a short space of time. For this reason, the maltitol is commercially available in about 75% aqueous solution. However, in such form, it is inconvenient to use compared with the above other sweetening agents.

According to the present invention, there is provided a process for the preparation of powdered maltitol which comprises adding a paste material as hereinafter defined suitable as food additive to an aqueous solution of maltitol, and then drying and, where necessary pulverizing, the product thus obtained to form a powder.

The paste materials of this invention are those which when mixed with water form a cohesive mixture.

The paste materials used in the present invention may be one or more of sodium alginate, propylene glycol alginate, sodium carboxymethylcellulose, sodium carboxymethylstarch, sodium starch phosphate, methylcellulose, hydroxyethyl starch, polyvinyl pyrrolidone, and polysaccharide of tamarind seed (Griloid; Trade name of Dainippon Pharmaceutical Co. Ltd. in Japan), which may be used independently or in combination. It is preferable to add 1.0 to 10.0 weight % or more of the paste materials based on the anhydrous maltitol. The upper limit of this addition is not particularly limited, but a maximum of about 10% of addition is preferable due to the decrease of the sweetness of the product.

The concentration of the maltitol in aqueous solution is not particularly limited, but it is preferable to adjust to 25 to 75% to prevent the loss of yield in the drying process.

The drying process may be carried out by conventional drying method such as, for example, spray drying or vacuum drying.

In one aspect of the invention, some advantage is noted in the drying process *per se*, that is for example, when maltitol only is dried by spray drying, it cannot be powdered because of the adhesion of the atomized maltitol in jelly state on the inner wall of the chamber, and when dried in vacuum, even if the dried mass obtained is pulverized, the powder changes to a gelatinous state in a very short time.

However, according to the method of the present invention by adding the paste materials to the maltitol, it simply forms a

power in the spray drying, and after vacuum drying the pulverization of the dried mass is very easy.

The powdered maltitol thus obtained has a great deal of fluidity, does not change to jelly state during long storage, may be utilized in a similar manner to the common sweetening agents, and is easily soluble in water.

The effect of the addition of the paste materials to the maltitol, which is illustrated by the "repose angle" which is the indication of the fluidity, can be shown from the following table. The "repose angle" is measured by gently pouring an amount of the product onto a horizontal surface to form a cone-shaped pile, and measuring the angle between the said surface and a side of the cone.

Paste Materials	Concentration of paste material Note 2	Repose Angle of Spray Dried Sample		Repose Angle of Vacuum Dried Sample	
		Just after Preparation	After Storage for 24 hrs. at 20% R.H. at 25 C.	Just after Preparation	After Storage 24 hrs. at 20% R.H. at 25°C.
No addition		impossible to measure Note 1	impossible to measure Note 1	45	impossible to measure Note 1
Sodium alginate	1.0	41	42	39	41
"	2.0	39	40	40	40
"	5.0	39	39	38	40
Propylene glycol alginate	1.0	35	38	40	41
"	2.0	32	35	41	41
"	5.0	31	35	40	40
Propylene glycol alginate + Sodium alginate	0.8 1.2	33	37	39	40
Sodium Carboxy-methylcellulose	1.0	40	41	38	39
"	2.0	41	41	40	42
"	5.0	41	41	40	42

Paste Materials	Concentration of paste material Note 2	Repose Angle of Spray Dried Sample		Repose Angle of Vacuum Dried Sample	
		Just after Preparation	After Storage for 24 hrs. at 20% R.H. at 25°C.	Just after Preparation	After Storage for 24 hrs. at 20% R.H. at 25°C.
Sodium Carboxy-methylstarch	1.0	42	43	42	45
"	2.0	39	41	35	39
"	5.0	39	40	34	38
Sodium starch phosphate	1.0	39	42	37	40
"	2.0	38	40	36	38
"	5.0	38	40	30	35
Methylcellulose	1.0	39	41	38	40
"	2.0	39	40	36	39
"	5.0	39	39	36	38
Hydroxyethyl starch	2.0	40	41	39	42
Polyvinyl-pyrrolidone	2.0	40	41	38	40
Polysaccharide of tamarind seed (Griloid (R))	2.0	38	39	37	38

Note 1: Impossible to measure due to the fact that the maltitol is obtained in jelly state or changes to the jelly state in measuring.

Note 2: Added amount of the paste materials are w/w % based on the anhydrous maltitol.

Note 3: The respective values are an average of ten experiments under the same conditions.

Note 4: R.H. means relative humidity.

As is evident from the above results, when the paste materials are not added to the maltitol, free flowing powdered maltitol is not obtained by spray drying, and even if, in the case of vacuum drying, the product obtained as a powder is stored in a low humidity, such as 20% relative humidity (at 25° C.), it changes to a jelly state in a short time.

On the other hand, the powdered maltitol obtained by the method of the present invention by adding the paste materials loses almost none of its fluidity when similarly stored, so that the effect of addition of the paste materials is remarkable.

As a comparison the repose angle of commercially available instant coffee powder and instant cocoa powder are 38° to 42° C, and 38° to 43°, respectively.

Following is a description by way of example only of methods of carrying the invention into effect.

Example 1.

Sodium alginate (75 g.) is added to 7.5 Kg. of 50% aqueous solution of maltitol, and is dissolved with stirring, and then the solution is dried with a spray dryer to obtain 3.7 Kg. of dried powder of maltitol. In this step, a centrifugal atomizer is used, and the condition of spray drying is as follows:

Inlet air temperature	100° to 130° C.
Outlet air temperature	50° to 75° C.
Supply of the solution	400 ml/min.
Blow rate	8 m^3/min.
Rotary speed of atomizer	25,000 rpm.

Example 2.

Sodium carboxymethylcellulose (75 g.) is added to 10 Kg. of 75% of aqueous solution of maltitol and is dissolved, and the solution is dried in vacuum by the conventional method as follows: hot plate temperature 60° C, for 3 hours. After the obtained dried mass is cooled, it is crushed with suitable pulverizer mounted in which was a sieve of suitable mesh to separate the powder.

Example 3.

Sodium alginate (45 g.) and 30 g. of propylene glycol alginate are added to 7.5 Kg. of 50% aqueous solution of maltitol and are dissolved with stirring, and the solution is dried with a spray dryer under the same conditions as those of Example 1 so as to obtain maltitol powder.

Example 4.

Sodium carboxymethyl starch (125 g.) is added to 10 Kg. of 25% aqueous solution of maltitol, and is dissolved, and the solution is dried with a spray dryer under the same condition as those in the Example 1 so as to obtain maltitol powder.

Example 5.

Hydroxyethyl starch (100 g.) is added to 10 Kg. of 50% of aqueous solution of maltitol, and is dissolved and the solution is dried in vacuum under the same conditions as those in the Example 2, and then the obtained mass is crushed so as to obtain the maltitol powder.

WHAT WE CLAIM IS:—

1. A process for the preparation of powdered maltitol which comprises adding a paste material as herein defined, suitable as a food additive, to an aqueous solution of maltitol, and then drying and, where necessary pulverizing, the product thus obtained to form a powder.

2. A process as claimed in Claim 1, wherein the amount of the paste materials is 1.0 to 10.0 weight % based on the anhydrous maltitol in the solution.

3. A process as set forth in Claim 1 or Claim 2, wherein the concentration of maltitol in aqueous solution is 25 to 75 weight %.

4. A process as set forth in any one of claims 1 to 3 wherein said drying step is performed using spray or vacuum drying.

5. A process as set forth in any one of the preceding claims wherein said paste material is one or more of sodium alginate, propylene glycol alginate, sodium carboxymethylcellulose, sodium carboxymethylstarch, sodium starch phosphate, methyl cellulose, hydroxyethyl starch, polyvinylpyrrolidone and polysaccharide of tamarind seed.

6. A process for the preparation of powdered maltitol substantially as described in any one of the specific Examples hereinbefore set forth.

7. Powdered maltitol whenever produced by the process claimed in any preceding claim

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